

# NIOSH Guidelines for Aerosol Sampling

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# Early days of aerosol sampling

- From the early 1900's, the U.S.A. used particle number measurements (konimeter, 1916; Greenburg-Smith impinger 1922; thermal precipitator, 1935)
- Limit values were expressed as millions of particles per cubic foot (counted under microscope)
- Most were area samples as personal pumps had not yet been invented
- Most samples were for mineral dusts in mining and quarrying



Konimeter, BGI photo library

# Impingers

- Air is drawn into a liquid (water) through a tube with a nozzle
- Particles are accelerated to collide with the base where they are stopped and wetted and retained
- Popular from development in the late 1920's (Greenburg-Smith impinger was later replaced by “midget impinger”) until the 1960's-80's
- Very fine particles are not collected efficiently
- Not preferred for personal sampling



# History continued

- Bureau of Mines first publishes on collecting airborne particles on glass-fiber filters in 1948
- In 1957, the USPHS publishes that they had been using filters in metal holders since 1953, and extolled their virtue for both counting and non-counting (e.g. gravimetric or chemical) analyses
- The plastic “Millipore Monitor” cassette developed for “clean-room” particulate monitoring in 1956 is featured in 1960 1<sup>st</sup> ed. ACGIH Air Sampling Instruments Handbook
- Used on top of the nylon Dorr-Oliver cyclone for respirable sampling

# Traditional filter holder: 37 mm closed-face cassette (CFC)

- Cheap (~\$1) and disposable
- Helps to prevent accidental or deliberate damage to the filter
- Can be used with an internal capsule
- Flow-rate: 1-4 liters/minute
- Does NOT sample  
“Total Dust”



4 mm  
Entry  
Orifice

# “Total Dust”

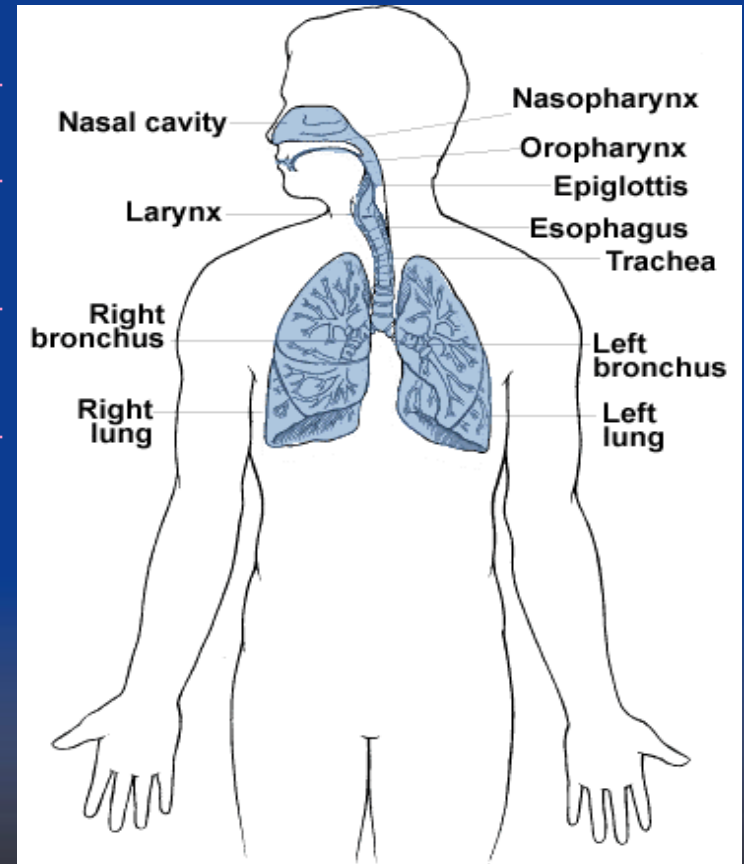
- One of the worst errors ever was to state that any sampler collected “Total Dust”
- Even isokinetic sampling probes have problems in sampling all particles sizes according to their actual concentration in the atmosphere
- But why would you want to? The human nose and mouth is not an isokinetic sampler either
- To estimate exposure accurately we should use a sampler that is relevant to biology

# Respiratory system

Nasopharyngeal  
Region

Tracheobronchial  
Region

Respirable  
Region

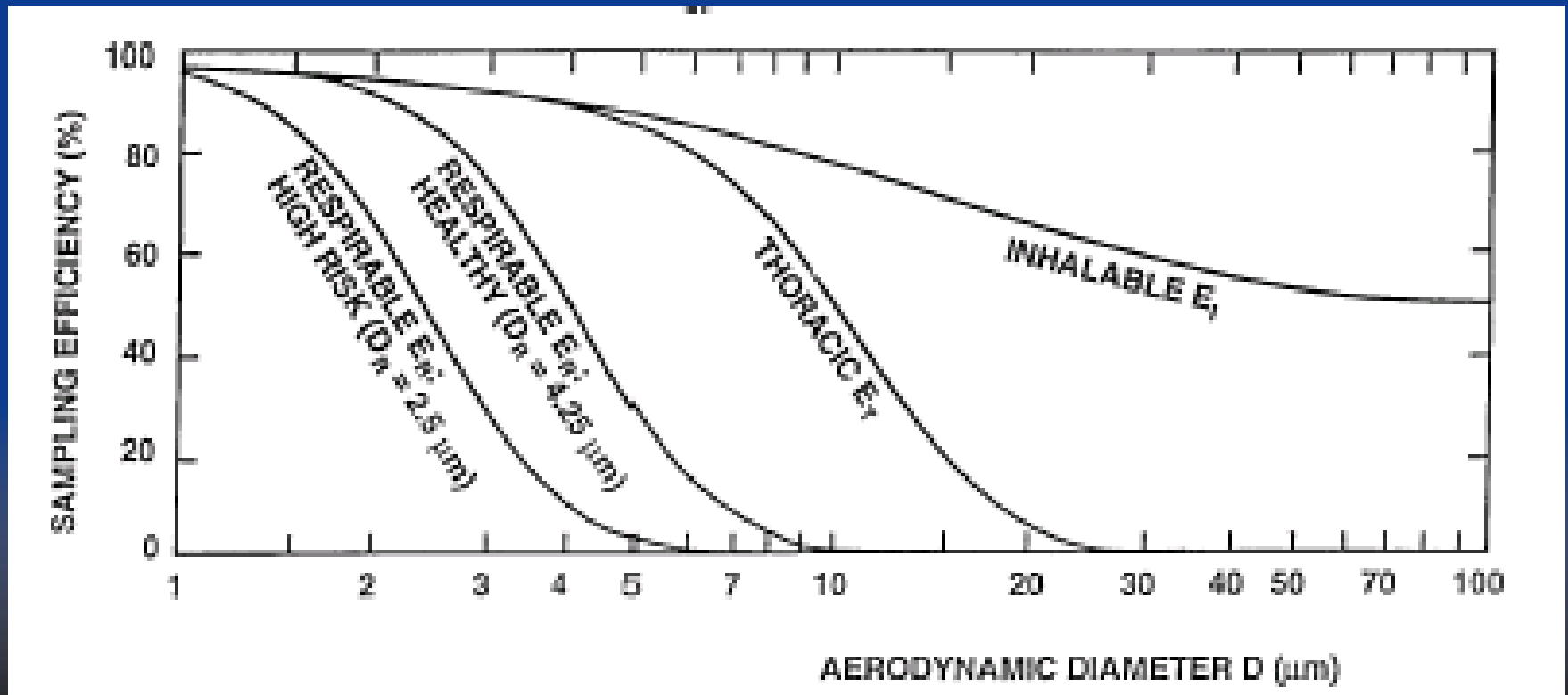


The deposition of particles in different regions of the lung has implications for disease – in the 1980's the International Organization for Standardization (ISO) published size distributions with relevance to health outcomes

# Size-selective sampling

- Particle size selective sampling has been recommended based on the likelihood of penetration of particles to different regions within the respiratory tract
  - ◆ “Inhalable”, “thoracic”, and “respirable” mass fractions were established by ISO (and ACGIH)
- The inhalable fraction was based on experiments with human subjects and samplers were calibrated against a “breathing” manikin “inhaling” particles of different sizes through its mouth and nose

# Size-selective sampling conventions (ISO TR 7708)



**Source: Air Sampling Instruments, ACGIH**

# ACGIH “inhalable” guidelines

- Particles Not Otherwise Classified (PNOC)
- Diatomaceous earth
- Wood dust
- Continuous filament glass fiber
- Ni, Mn, Be, Mo, Ti, and most compounds
- Mg and V oxides
- Calcium sulfate
- Carbon black
- Flour dust
- Asphalt fume (benzene-solubles)
- Natural rubber latex
- Mineral oil
- Many pesticides
- Toluene di-isocyanate (TDI)

# Are there “inhalable” samplers?

- ISO and ACGIH recommend that a particular size selective convention be measured using a sampler whose performance matches the particle penetration probability of the convention
  - ◆ Thus an inhalable sampler should have performance matching the inhalable convention
- In the 1980's in Europe many different samplers for “total dust” formed the basis of the different national standards
- A study was funded to determine which, if any, met the inhalable convention

# Samplers tested for “inhalability” in major European study

- 37mm Closed-face cassette (Spain & USA)
- 37mm Open-face cassette (Sweden)
- CIP10-I (France)
- PAS-6 (Netherlands)
- PERSPEC (Italy)
- GSP (Germany)
- IOM (United Kingdom)
- Seven-hole (United Kingdom)

L Kenny et al., *Ann Occup Hyg* 41: 135 (1997)

# Results of inhalability testing

Sampler		Calibration Constant
IOM	BIAS <50%	0.95
37 mm CFC	BIAS >50%	1.49

*DL Bartley Appl Occup Environ Hyg 13: 274 (1996)*

Important: Internal wall deposits were not accounted for in evaluating the closed-face cassettes (only the filter catch was analyzed) while internal wall deposits were included in the analysis of the IOM samplers

# 37 mm CFC usage at the time

- OSHA has always used the internal “flying saucer” for gravimetric analysis, but only discussed the inclusion of visible deposits in other methods until recently (now updated to include all samples)
- NIOSH NMAM 0500 only mentions analyzing the filter catch of the 37 mm CFC
  - ◆ However, the “Blue Pages” Chapter 0, Part 7 (Paul Baron) discussed the possibility of deposits on internal surfaces of the cassettes and recommends they be included as part of the sample

# IOM Sampler

- Institute of Occupational Medicine (IOM) personal inhalable sampler
- Designed in wind tunnel studies to approximate the inhalable convention
- Internal deposits were noted during development and were considered to be an integral part of the sample
- 2 liters/min



15 mm  
Entry Orifice

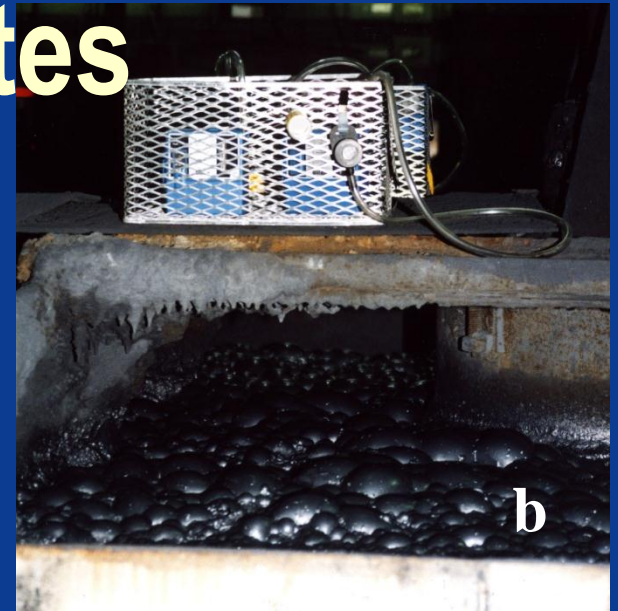
# Converting from filter-only CFC results to inhalable fraction as measured by the IOM sampler

TYPE OF AEROSOL	FACTOR
Dust - powder handling, mining, grinding, etc.	2.5
Mist - paint spray, oil mist, electroplating, etc.	2.0
Hot process - smelting, refining, foundries, etc.	1.5
Smokes and fumes - includes welding	1.0

# NIOSH Metals study sites



a



b



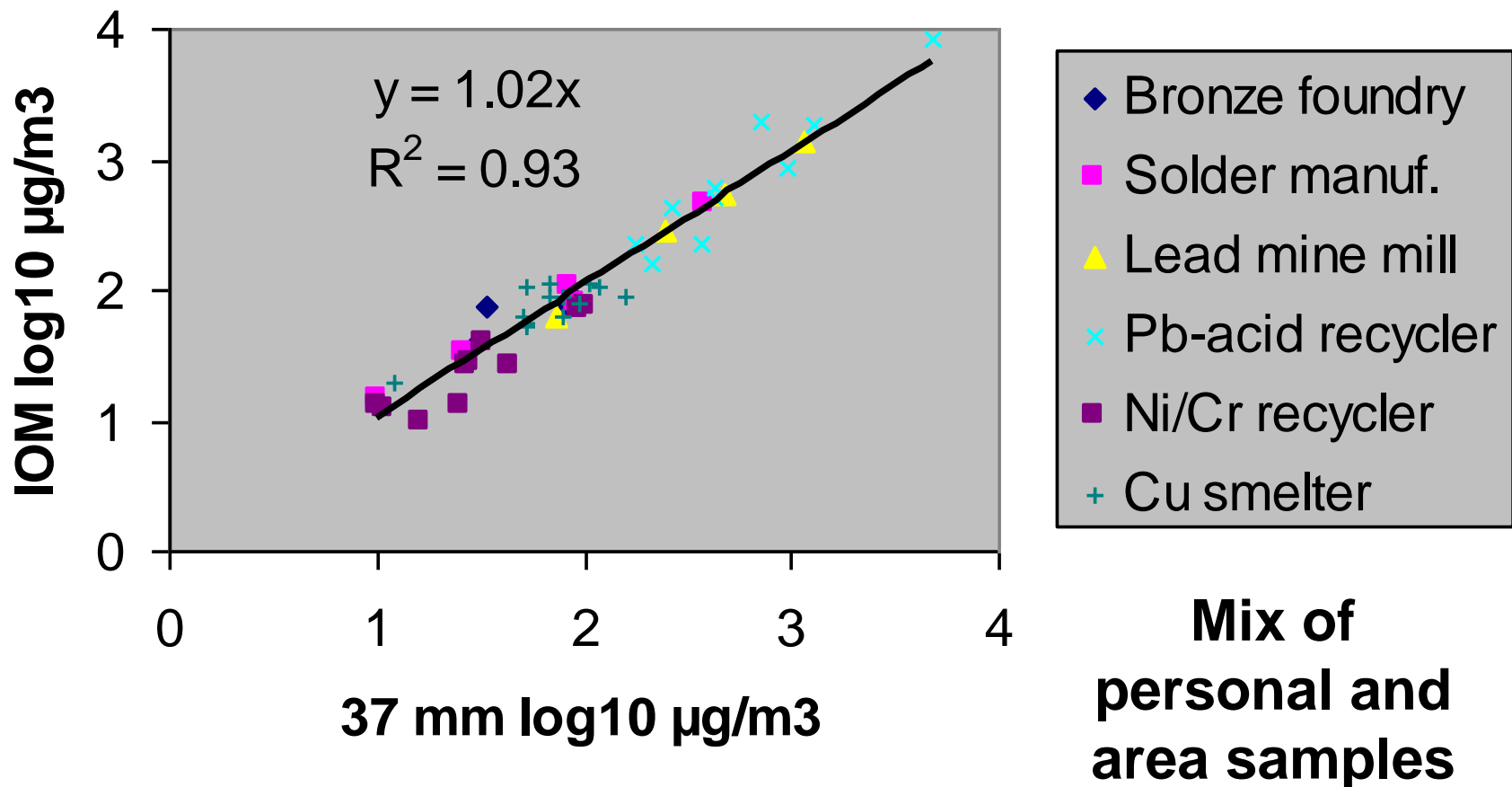
d

a. Pb battery recycler  
b. Pb ore concentrator  
c. Solder manufacturer  
d. Bronze foundry  
Also Ni - Cr recycler  
and Cu smelter (not  
shown)



c

# Comparison of IOM and CFC filters-only (51 pairs)



# Conclusion of NIOSH study

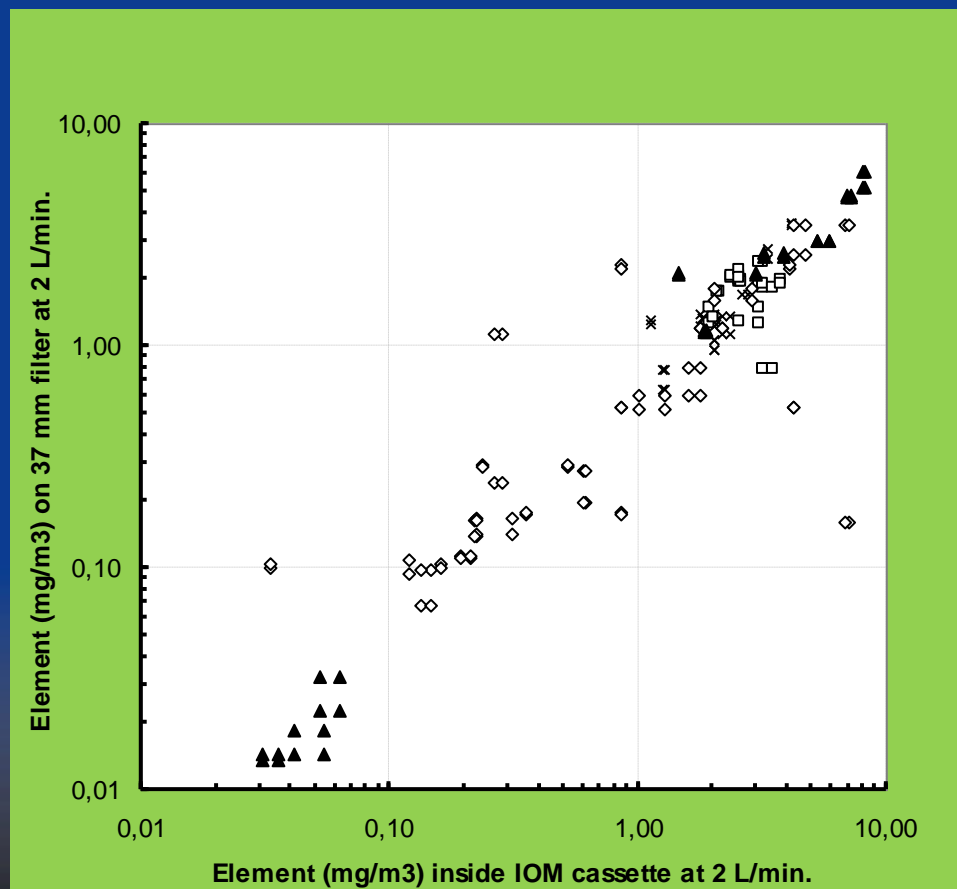
- No significant difference was observed in the metals found on the filters of CFC and IOM samplers side-by-side
  - ◆ Why not? - A difference was expected
- Perhaps it was because we did not account for the internal surface deposits in the IOM
- The IOM and CFC internal surface deposits were analyzed in some samples from some of the sites
- These data were included with similar data from relevant studies by others in metals industries (references available on request)

# CFC Median non-filter deposits

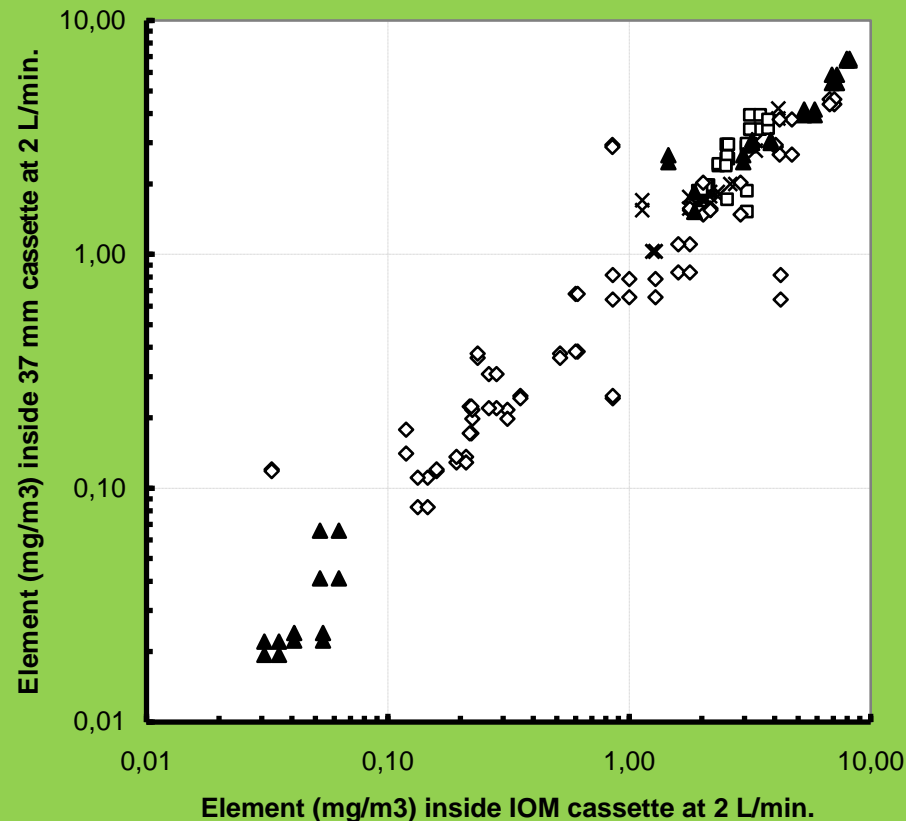
Environment	n	Agent	Median wall deposit
Cu smelter	18	Cu	<b>21%</b>
Pb ore mill	9	Pb	<b>19%</b>
Solder manufacture	30	Pb	<b>29%</b>
Battery production	16	Pb	<b>28%</b>
Welding	10	Cr(VI)	<b>5%</b>
Plating	12	Cr(VI)	<b>12%</b>
Paint spray	29	Cr(VI)	<b>7%</b>
Foundry	9	Zn	<b>53%</b>
Zn plating	18	Zn	<b>27%</b>
Cast iron foundry	18	Fe	<b>22%</b>
Grey iron foundry	18	Fe	<b>24%</b>
Bronze foundry	6	Cu, Pb, Sn, Zn	<b>19%, 13%, 0%, 15%</b>
Cuproberyllium	4	Cu, Be	<b>31%, 12%</b>

# Relationship of CFC (filter-only) and IOM cassette (filter plus walls)

M Demange et al.  
“Field comparison of  
37-mm closed-face  
filter cassettes and  
IOM samplers”  
*Appl Occup Environ  
Hyg* 17: 200 (2002)

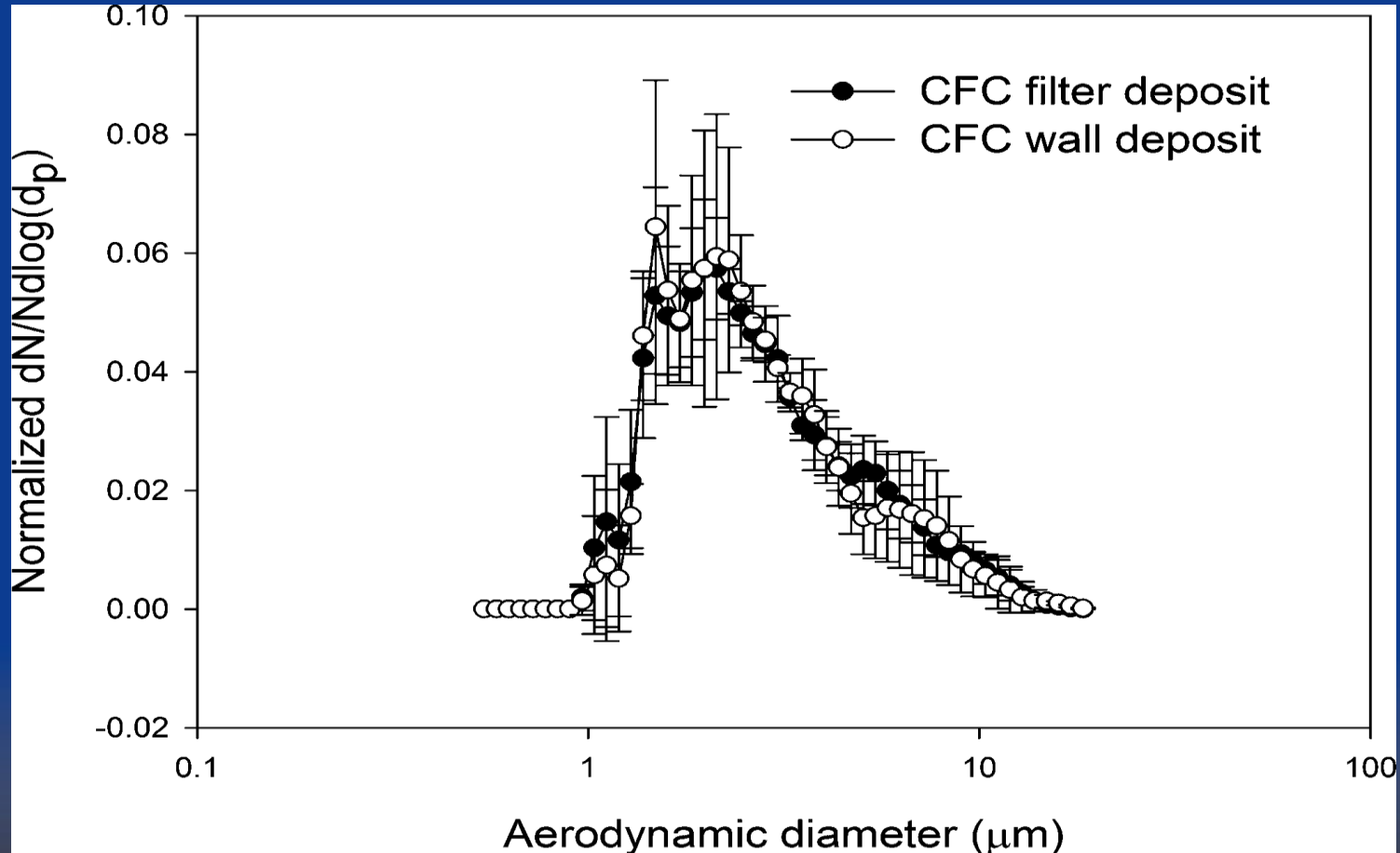


# After adding CFC wall deposits



× E2 (Fe - Cast iron machining and welding)    □ E3 (Fe - Grey cast iron machining)    ◇ E4 (Pb - Lead and Zinc refinery)    ▲ E5 (Ni - Nickel refinery)

# Size distributions: wall vs. filter



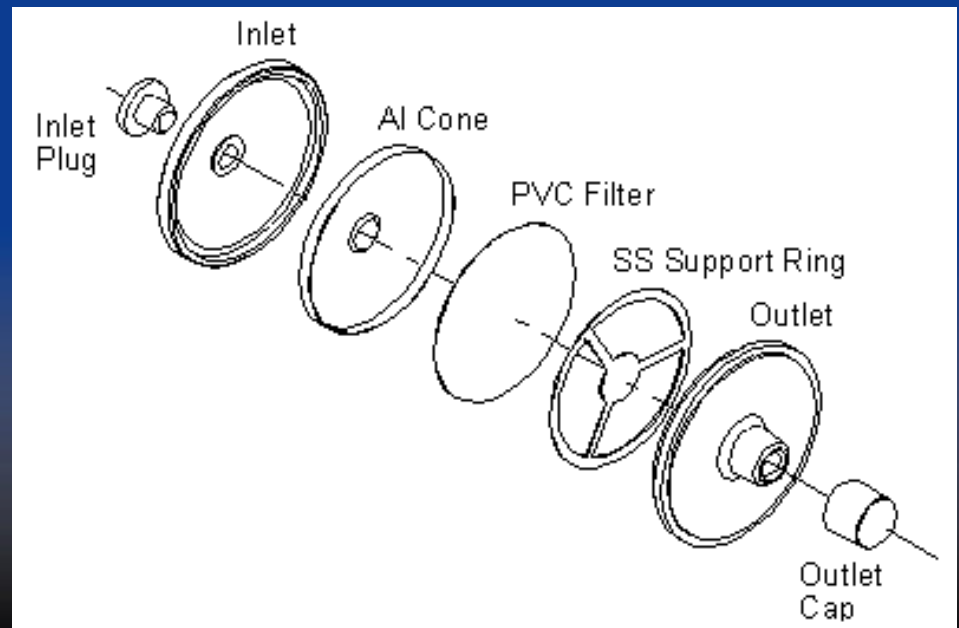
T Lee et al., *Aerosol Sci Technol* 43: 1042 (2009)  
See also T Lee et al., *Aerosol Sci Technol* 46: 411 (2011)

# Conclusions

- Particles that enter the CFC don't always end up on the filter ("wall deposits" includes under cap)
- The proportion on the inner surface of the cassettes can be high even when not visible
- There is little difference in particle size from the filter deposit to suggest they would not also enter the nose and mouth of a breathing person
- Including them in the analysis gives results closer to those of a sampler operating in accordance with the "inhalable" convention
- So how should they be included?

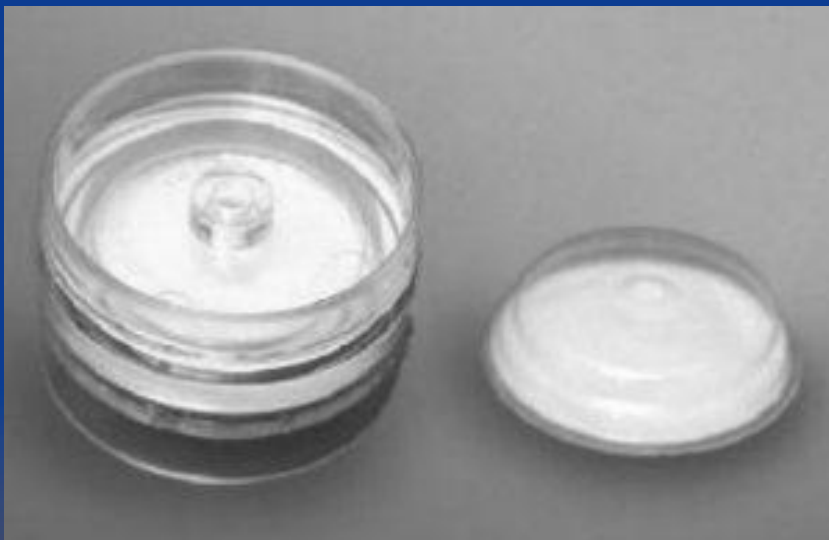
# OSHA gravimetric sampling

OSHA gravimetric method: PVC-Aluminum foil capsule (“flying saucer” sampler) already in use before 1970; adopted by OSHA and MSHA for gravimetric analysis (OSHA method PV 2121)

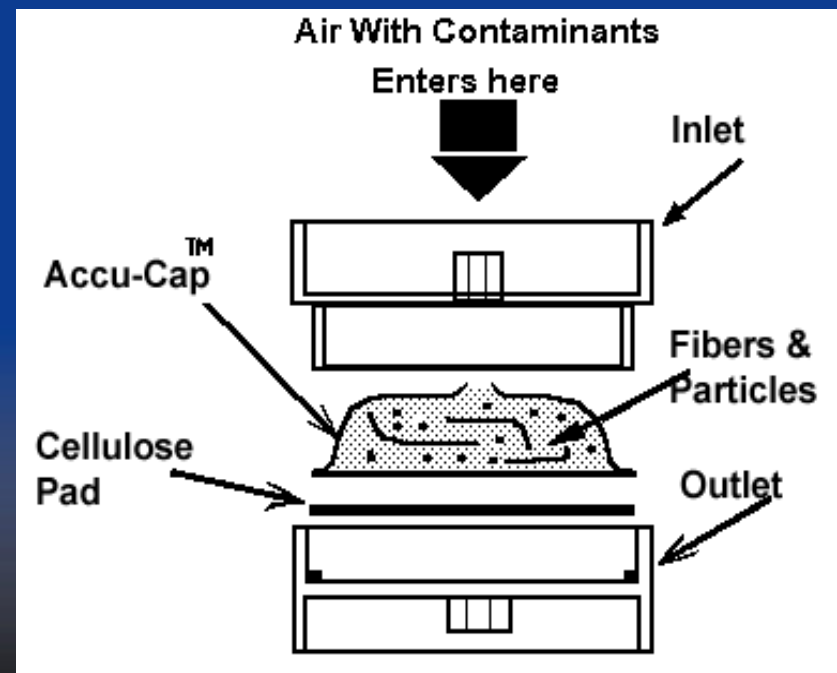


# Accu-cap™

Based on MA Puskar et al. "Internal wall losses of pharmaceutical dusts during closed-face, 37-mm polystyrene cassette sampling"  
*Am Ind Hyg Assoc J* 52: 280 (1991)



(Courtesy of SKC, Inc. Omega Specialty Division)

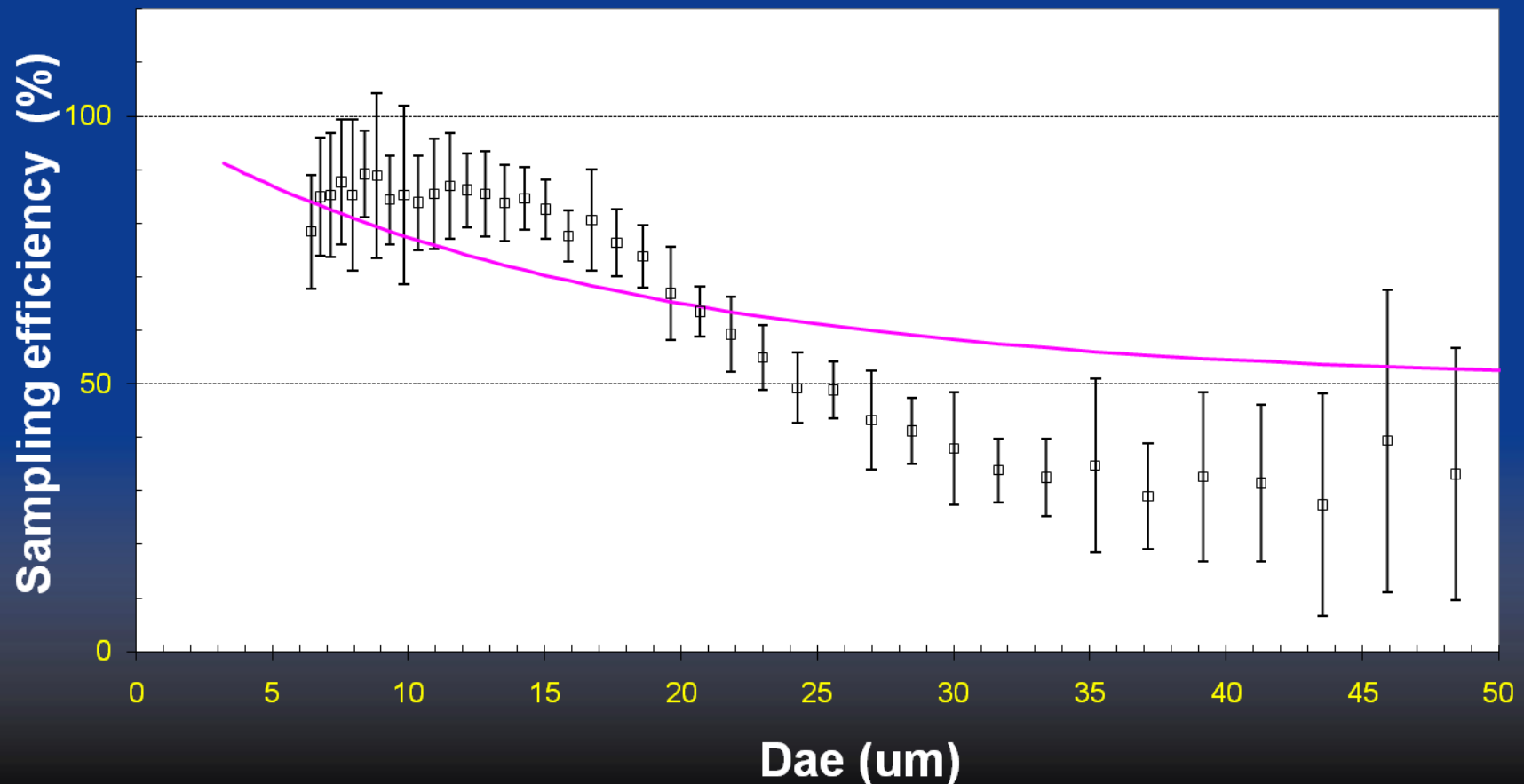


# Is the CFC (inc. wall deposits) an “inhalable” sampler?

- Aerosol science tells us that the higher velocity through the 4 mm CFC inlet would not sample very large particles as well as the lower velocity through the 15 mm inlet of the IOM
  - ◆ This is borne out in laboratory tests where particles larger than 20  $\mu\text{m}$  aerodynamic diameter are not sampled so well
- However, two questions remain:
  - ◆ Are there very large particles in the environment?
  - ◆ If they are under-sampled, is it significant?

# French (INRS) laboratory test data

Accu-cap flow 2l/min, on bluff body turning twice/min,  
1m/s wind speed



# NIOSH wood dust study

- Wood dust represents about the largest particles likely found in the occupational environment
  - ◆ Very little mass < 10  $\mu\text{m}$  Aerodynamic Equivalent Diameter; particles can be > 100  $\mu\text{m}$  AED
- NIOSH study of IOM samplers and PVC Accu-caps in 37 mm CFC (plus some other samplers)
  - ◆ Side-by-side samples in 7 different workplaces
  - ◆ IOM:Accu-cap average ratio was 1.56:1
  - ◆ With natural side-to-side variation this was not significantly more than a 35% difference (considered equivalence) with only 32 pairs
  - ◆ T Lee et al., *Ann Occup Hyg* 55: 180 (2011)

# Removing wall deposits for chemical analysis: rinsing

Metal (n=10)	Quartz fiber filters	Surfaces (H <sub>2</sub> O rinses)	Surfaces (after H <sub>2</sub> O rinse)
Cd	72	10	18
Fe	67	12	21
Pb	74	12	14
Zn	75	10	15

K Ashley et al., *J Anal At Spectrom* 16: 1147 (2001)

# Removing wall deposits: wiping

Mass 1 <sup>st</sup> wipe ( $\mu\text{g Pb}$ )	Mass 2 <sup>nd</sup> wipe ( $\mu\text{g Pb}$ )
1	1
18	5
82	7
99	6
210	8
320	5
490	7
650	11
730	25



**Demonstrates  
adequacy of  
single  
wiping**

**W Hendricks et al.,  
*JOEH* 6: 732 (2009)**

# Removing wall deposits for chemical analysis

- A metal or PVC capsule is not suitable for acid digestion – the alternative is wiping
  - ◆ 10% nitric acid rinse was not effective at removing all wall deposits (OSHA)
  - ◆ a single wipe of the interior was effective in removing the deposit in the majority of cases
- OSHA uses a “smear tab” or 1 x 2 inch portion of “Ghost” wipe, moistened with deionized water; cellulose wipes likely give better matrix and recovery
- Same issue applies to IOM or other samplers where wall deposits need to be included!

# Alternatives to rinsing and wiping

- A cellulosic capsule is suitable for acid digestion – tested in all ISO Standard alternative procedures
  - ◆ Measurable background levels of Ca, Mg and P, but not significant in relation to OELs for e.g.,  $\text{Ca(OH)}_2$ , MgO or  $\text{H}_3\text{PO}_4$
  - ◆ Below or near detection limit for 25 other elements
- Interlaboratory Studies (ILS) according to ASTM Standards performed on prototypes from SKC, Inc.
- Thinner “Solu-Serts” are now available from Air Sampling Devices, LLC (less mass giving lower background)
- Cellulosic capsules are NOT suitable for weighing

# Interlaboratory study of digestible capsules (Pb)

<i>Sample matrix</i>	<i>Ref. , μg Pb</i>	<i>Interlab mean, μg Pb</i>	<i>Std. dev.</i>	<i>% Recovery (% RSD)</i>
Liquid spike (low)	18.0	18.2	1.0	101 (5.0)
Liquid spike (high)	42.1	37.5	1.9	89.1 (4.7)
Soil (low)	22.1	21.6	2.4	97.7 (10.2)
Soil (high)	54.5	49.8	2.6	91.3 (4.8)
Paint (low)	21.3	22.2	0.5	104 (1.9)
Paint (high)	51.8	49.2	1.3	95.0 (2.4)

M Harper & K Ashley, *J Occup Environ Hyg*  
9: D125 (2012)

# ILS with multielement aerosol-dosed digestible capsules (n = 8)

Element / level (µg)	Mean ± Std. Dev. (µg/m <sup>3</sup> )	RSD (%)
Cd (1.5)	6.0 ± 0.79	13
Cr (350)	290 ± 14	4.9
Co (0.3)	1.5 ± 0.006	4.0
Cu (30)	140 ± 6.2	4.5
Fe (750)	590 ± 46	7.8
Pb (75)	58 ± 4.2	7.3
Mn (30)	130 ± 11	8.4
Ni (15)	67 ± 7.1	11

M Harper & K Ashley, *JOEH*, in “Latest Articles” to be published in next issue

# In-cassette digestion

- French national method for metals using glass-fiber filters and ultrasonic digestion with nitric and hydrofluoric acids
  - ◆ Note: microwave digesters that can easily accommodate 37 mm CFC's do not exist
- French have also used MCE filters with PVC Accu-caps – allows weighing of filters prior to digestion – perchloric acid is used to dissolve filter
- Reasonable approaches, but neither is likely acceptable to US laboratories in their current form
- More research is needed – in-cassette digestion has been found to work in other methods

# Wall deposits in respirable sampling

- Cyclones often attached to CFC's holding filters
- AIHA Proficiency Analytical Testing (PAT) samples for respirable crystalline silica are sampled from a chamber with cyclones and cassettes
- L Dobson et al., "Evaluation of Quartz Residue on Cassette Interiors of AIHA Proficiency Samples" J ASTM Int, 2: paper JAI12229, on-line (2005)
- Half the PAT samples had detectable wall deposits, 7.7 – 26% of total
- Field samples: 2-pc up to 32%, 3-pc up to 55%

# NIOSH Methods with recommendation to account for wall deposits:

## 1. Gravimetric analysis

Methods	Recommended techniques
0500 – Particles (“total”) 5000 – Carbon black	Use weight-stable internal capsule (e.g., PVC)
0600 – Particles (respirable)	<ol style="list-style-type: none"><li>1. Use static-dissipative (“conductive”) cassette</li><li>2. Use internal capsule (e.g., PVC)</li></ol>

# Affected NIOSH Methods:

## 2. Elemental analysis

Methods	Recommendations	Alternatives
<u>AA methods:</u> 7013 – Al; 7024 – Cr; 7027 – Co; 7029 – Cu; 7030 – Zn; 7046 – Ba; 7048 – Cd; 7074 – W; 7082 & 7105 – Pb; 7102 – Be; 7900 – As  <u>ICP-AES:</u> 7300 series – Elements	1. Wipe internal non-filter surfaces; add wipe to filter & digest together  2. Use digestible internal capsule (i.e., cellulosic)	Rinse internal non-filter surfaces with dilute acid (must first demonstrate effectiveness)
7020 – Ca 7701 – Pb 7704 – Be	Wipe internal walls & add wipe to filter for extraction	Perform within-cassette extraction

# Affected NIOSH Methods:

## 3. Cr(VI) & Alkaline dusts

Methods	Recommendations
7600, 7605, 7703 – Cr(VI) 7401 – Alkaline dusts	<ol style="list-style-type: none"><li>1. Wipe internal surfaces &amp; extract along with filter</li><li>2. Within-cassette extraction or rinsing</li></ol>

## 4. Internal deposits already addressed in method

Methods	Recommendations
5005 – Thiram; 5011 – Ethylene thiourea; 5030 – Cyanuric acid; 5032 – Pentamidine isethionate	Rinse internal surfaces with extract solution & add to filter
5700 – Formaldehyde (textile or wood dust)	Use IOM inhalable sampler

# Current NIOSH research efforts

- Revising affected NIOSH aerosol sampling methods (e.g., gravimetric & elemental anal.)
- Consider other NIOSH methods potentially affected (e.g., silica; metalworking fluids; asphalt fume; isocyanates)
- Ensure consistency with voluntary consensus standards (ASTM & ISO)
- Collaboration with international agencies (e.g., MOU's with IFA, Germany and HSL, UK)

# Acknowledgments (1)

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- HSL – Buxton, United Kingdom
- INRS – Nancy, France
- IRSST – Montréal, Canada
- NIOH – Oslo, Norway
- OSHA SLTC – Sandy, UT
- RTI Int'l – Research Triangle Park, NC
- SRNL – Savannah River Site, SC
- WOHL – Madison, WI

# Disclaimer

Statements in this presentation regarding NIOSH Methods do represent the policy of NIOSH.

However, mention of commercial devices should not be construed to indicate that other devices are not fit for the same purpose. Mention of ACGIH or its products does not constitute endorsement by the US government. Authors have made every attempt to ensure the accuracy of all information but do not warrant it free from error or omission.

**Beryllium  
borate  
mineral –  
newly  
acquired by  
the  
Smithsonian**

